

# Novel (4,8)-connected scu Framework Constructed by Tetrakis(4-benzoic acid)ethylene

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Supporting Information

## Experimental Section

All the starting materials were commercially available reagents for analytical grade and used without further purification.

**Tetrakis(4-benzoic acid)ethylene·0.5H<sub>2</sub>O (H<sub>4</sub>tbe·0.5H<sub>2</sub>O):** To a solution of tetrakis(4-bromophenyl)ethylene (8.38g, 12.9mmol) in 300 mL of dry THF, n-butyllithium (2.5 M in hexane, 26 ml, 65.0 mol) was added dropwise at -78 °C under N<sub>2</sub>. After addition, the mixture was stirred for another 2 h at -78 °C, and solid carbon dioxide (100 g) was added in portions. The mixture was warmed to room temperature and stirred overnight. The solution was adjusted to pH = 1 with conc. HCl (15 mL) and extracted by mixed solvent of ether and THF to give white solid. A mixture of the white solid, conc. H<sub>2</sub>SO<sub>4</sub> (8.0 mL) and ethanol (250 mL) in a 500 mL three-necked flask was stirred at reflux for 20 h. The resulting solution was concentrated under reduced pressure. The residue was purified by silica gel chromatography to yield 2.62 g (38.6%) of tetraester a white solid. A mixture of

tetraester, sodium hydroxide (3.43 g), ethanol (150 mL) and H<sub>2</sub>O (150 mL) in a 500 mL three-necked flask was stirred at reflux for 20 h. The resulting solution was concentrated under reduced pressure, and the residue was acidified with conc. HCl at 0°C. The precipitate was collected and dried to yield 2.01 g (94%) of tetrakis(4-benzoic acid)ethylene·0.5H<sub>2</sub>O as a white solid. Anal. Calcd for C<sub>30</sub>H<sub>25</sub>O<sub>10.5</sub> (%):C, 65.09; H, 4.55. Found (%):C, 65.31; H, 4.35. Selected IR (KBr, cm<sup>-1</sup>): 3444.4(m), 3175.6(w), 1693.1(s), 1605.8(s), 1410.3(m), 1248.3(m), 1177.8(m), 1105.0(m), 748.6(m).

Table S1 Selected bond lengths (Å) and angles (deg) for **1**.

Compound <b>1</b>					
Co1-O4	2.099(2)	Co1-O3	2.108(17)	Co1-O2b	2.087(2)
Co1-O2c	2.087(2)	Co1-O1	2.101(2)	Co1-O1a	2.101(2)
Co1-O3'	2.161(17)	Co1-O3'a	2.161(17)		
O4-Co1-O3	174.7(5)	O4-Co1-O2b	87.5(3)	O3-Co1-O2b	96.1(3)
O4-Co1-O2c	87.5(3)	O3-Co1-O2c	96.1(3)	O2b-Co1-O2c	90.4(4)
O4-Co1-O1a	85.7(3)	O3-Co1-O1a	90.7(3)	O2b-Co1-O1a	173.2(3)
O2c-Co1-O1a	88.4(3)	O4-Co1-O1	85.7(3)	O3-Co1-O1	90.7(3)
O2b-Co1-O1	88.4(3)	O2d-Co1-O1	173.2(3)	O1a-Co1-O1	92.0(4)

Symmetry code : a: x, y, -z+2 b: x-1/2, -y+3/2, z c: x-1/2, -y+3/2, -z+2